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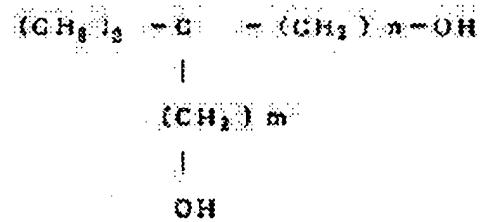
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(54) AQUEOUS INK CONTAINING BLACK PIGMENT, INK-JET RECORDING USING THE SAME AND APPARATUS FOR INK-JET RECORDING

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain an aqueous ink containing a black pigment, excellent in issuing stability after the interruption of a printing operation when used for an ink-jet recording, by compounding a specific compound, water and a coloring material comprising a specified self-dispersion type carbon black.

SOLUTION: This aqueous ink containing a black pigment is obtained by compounding (A) 0.1-15 wt.% of a coloring material comprising a self-dispersion type carbon black to whose surface one or more hydrophilic groups are bonded directly or through one or more other atomic groups, (B) 1-30 wt.% of a compound of the formula [(n) is 1 or 2; (m) is 0-2] (for example, 3-methyl-1,3- butanediol) and (C) 50-95 wt.% of water. The hydrophilic groups in the component A preferably comprise -COOM [M is H, an alkali metal or (organic) ammonium], -SO₃M, -SO₂NH₂, -PO₃HM, -PO₃M₂, -NH₃⁺, etc., and the other atomic groups in the component A preferably comprise a 1-12C alkyl, (substituent-containing) phenyl, (substituent-containing) naphthyl, etc.



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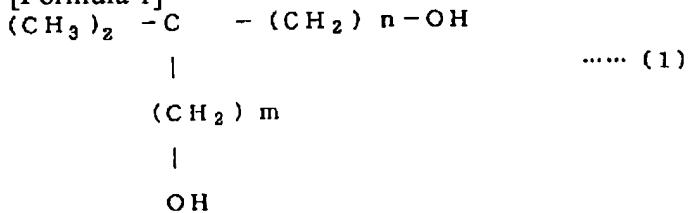
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CLAIMS

[Claim(s)]

[Claim 1] Aquosity black pigment ink characterized by containing the color material which consists of self-distributed carbon black with which at least one sort of hydrophilic radicals were combined through direct or other groups on the surface of carbon black, the compound expressed with the following general formula (1), and water.

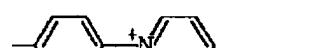
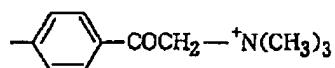
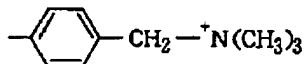
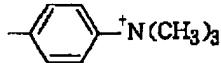
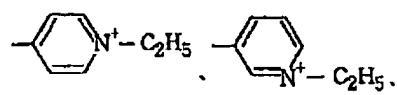
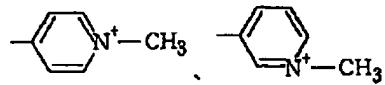
[Formula 1]

(However, the inside n of a formula expresses the integer of 1-2, and m expresses the integer of 0-2.)

[Claim 2] Aquosity black pigment ink according to claim 1 said whose hydrophilic radical is at least one sort chosen from the following hydrophilic radical and whose atomic group besides the above is the naphthyl group which may have the phenyl group which may have the alkyl group of the carbon atomic numbers 1-12, and a substituent, or a substituent.

[Formula 2]

– COOM, – SO₃M, – SO₂NH₂,
 – PO₃HM, – PO₃M₂, – SO₂NHCOR,
 – NH₃⁺, – NR₃⁺,



(However, as for the inside M of a formula, a hydrogen atom, alkali metal, ammonium, or organic ammonium is expressed, and R expresses the naphthyl group which may have a kill radical and a substituent with the carbon atomic numbers 1-12, and may have a phenyl group or a substituent more.)
 [Claim 3] Aquosity black pigment ink according to claim 1 or 2 whose content of the compound expressed with a general formula (1) is 1 - 30 % of the weight to the aquosity black pigment ink whole quantity.

[Claim 4] Aquosity black pigment ink given in any 1 term of claim 1 whose compound expressed with a general formula (1) is 3-methyl-1,3-butanediol - claim 3.

[Claim 5] Aquosity black pigment ink given in any 1 term of claim 1 whose compound expressed with a general formula (1) is 2 and 2-dimethyl-1,3-propanediol - claim 3.

[Claim 6] The ink jet record approach that this ink is characterized by being aquosity black pigment ink given in any 1 term of claim 1 - claim 5 in the ink jet record approach which give energy to ink, and this ink is made to fly as an ink droplet, and records on record material.

[Claim 7] The ink jet record approach according to claim 6 that said energy is heat energy.

[Claim 8] The ink jet record approach according to claim 6 that said energy is mechanical energy.

[Claim 9] The record unit to which this ink is characterized by being aquosity black pigment ink given in any 1 term of claim 1 - claim 5 in the record unit possessing the ink hold section which held ink at least, and the head section for making this ink breathe out as an ink droplet.

[Claim 10] The ink cartridge to which this ink is characterized by being aquosity black pigment ink given in any 1 term of claim 1 - claim 5 in the ink cartridge equipped with the ink hold section which held ink.

[Claim 11] The ink jet recording device with which this ink is characterized by being aquosity black pigment ink given in any 1 term of claim 1 - claim 5 in the ink jet recording device possessing the record unit which has the ink hold section which held ink at least, and the head section for making this ink breathe out as an ink droplet according to an operation of heat energy.

[Claim 12] The ink jet recording device with which the above-mentioned ink is characterized by being aquosity black pigment ink given in any 1 term of claim 1 - claim 5 in the ink jet recording device

possessing the ink cartridge equipped with the ink hold section which held ink at least, and the recording head for making this ink breathe out as an ink droplet according to an operation of heat energy.

[Claim 13] The ink jet recording device according to claim 12 which has the ink feed zone which supplies the ink held in the ink cartridge to the recording head section.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]**[0001]**

[Field of the Invention] This invention relates to the ink jet record approach and ink jet recording apparatus using aquosity black pigment ink and this ink using the carbon black suitable for ink jet record as a color material in more detail about aquosity black pigment ink, the ink jet record approach, and an ink jet recording apparatus.

[0002]

[Description of the Prior Art] Although the carbon black which was excellent in robustness etc. as a black coloring agent of printing ink is used widely conventionally, in order to use carbon black as a color material of water color ink, it is required that carbon black should be stabilized and distributed in an aquosity medium. Generally, since dispersibility of carbon black is not good, in order to acquire a homogeneity dispersed system, the method of adding a dispersant and distributing carbon black in an aquosity medium is taken.

[0003] On the other hand, when using ink for ink jet record generally, to become a stable drop from a hole with a detailed ink jet recording head (nozzle), and to be breathed out is demanded. When ink evaporates in the nozzle section of an ink jet recording head in coincidence, it is necessary for ink not to solidify. However, since it is hard to remelt after the resin which forms a dispersant adheres to an orifice etc. when the ink which the above-mentioned dispersant contained is used for ink jet record, blinding, the non-regurgitation of a drop, etc. may arise. Moreover, since the watercolor pigment ink containing a dispersant was viscous, when it performed the continuation regurgitation and high-speed printing covering long duration, it had the problem that the regurgitation became unstable or smooth record became difficult.

[0004] Then, in order to solve these problems, to JP,5-186704,A or JP,8-3498,A, the self-distributed carbon black which stability can be made to distribute is volatilizing by introducing a water-soluble radical on the surface of carbon, without using a dispersant. However, when the pause of printing for the nozzle corresponding to a null is completed during printing of the document and image which have a null also when the watercolor pigment ink using the self-distributed carbon black which does not use a dispersant as ink for ink jet record is used, there is a problem of the regurgitation of the beginning at the time of a restart being confused, or becoming the non-regurgitation.

[0005]

[Problem(s) to be Solved by the Invention] Then, the purpose of this invention is to offer the ink jet record approach and ink jet recording device using aquosity black pigment ink excellent in the regurgitation stability after a printing pause (for it to be henceforth called intermittent regurgitation stability), and this aquosity black pigment, when the conventional problem which was described above is solved and is used especially for ink jet record.

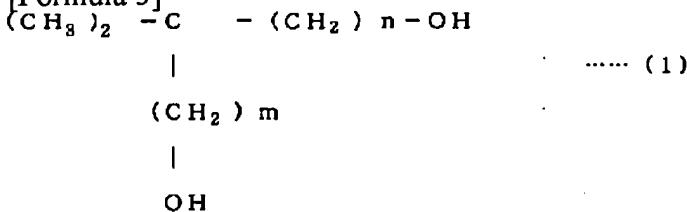
[0006]

[Means for Solving the Problem] The aquosity black pigment ink of this invention is characterized by containing the color material which consists of self-distributed carbon black with which at least one sort

of hydrophilic radicals were combined through direct or other groups on the surface of carbon black, the compound expressed with the following general formula (1), and water.

[0007]

[Formula 3]



(However, the inside n of a formula expresses the integer of 1-2, and m expresses the integer of 0-2.) The ink jet record approach of this invention is characterized by this ink being aquosity black pigment ink of above-mentioned this invention in the ink jet record approach which give energy to ink, and this ink is made to fly as an ink droplet, and records on record material.

[0008] The ink hold section in which the ink jet recording device of this invention held ink at least, In the ink jet recording device possessing the record unit which has the head section for making this ink breathe out as an ink droplet according to an operation of heat energy Whether this ink is aquosity black pigment ink of above-mentioned this invention; Or it sets to the ink jet recording device possessing the ink cartridge equipped with the ink hold section which held ink at least, and the recording head for making this ink breathe out as an ink droplet according to an operation of heat energy. It is characterized by this ink being aquosity black pigment ink of above-mentioned this invention.

[0009] According to this invention, especially when it uses for ink jet record, aquosity black pigment ink excellent in intermittent regurgitation stability is offered.

[0010]

[Embodiment of the Invention] Next, the gestalt of desirable operation is mentioned and this invention is explained more to a detail.

[0011] First, the self-distributed carbon black by which at least one sort of hydrophilic radicals were combined with the front face through direct or other groups as a color material is used. Consequently, the dispersant for distributing carbon black like conventional ink becomes unnecessary. As self-distributed carbon black used by this invention - SO₂ NH₂, -SO₂ NHCOR (R expresses the naphthyl group which may have the phenyl group which may have the alkyl group of carbon numbers 1-12, and a substituent, or a substituent.) etc. -- although the nonionic self-distributed carbon black which combined the substituent with the front face is sufficient, what has especially ionicity is desirable and what was charged in anionic, and the thing charged in cationicity are suitable.

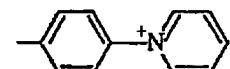
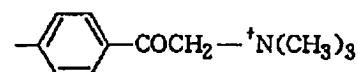
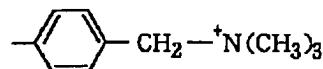
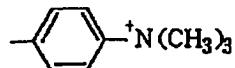
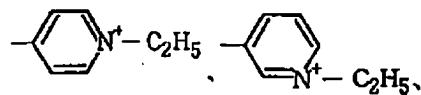
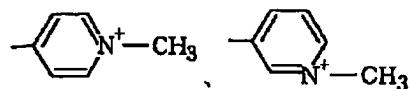
[0012] As a hydrophilic radical combined with the carbon black front face charged in anionic, -COOM, -SO₃ M, -PO₃ HM, -PO three M₂, etc. are mentioned, for example (however, M in a formula expresses a hydrogen atom, alkali metal, ammonium, or organic ammonium.). Especially in this invention, it is desirable to use that by which -COOM and -SO₃ M were combined with the carbon black front face in these.

[0013] Moreover, as an alkali metal, a lithium, sodium, a potassium, etc. are mentioned and, as for "M" in the above-mentioned hydrophilic radical, monochrome thru/or trimethylammonium, monochrome or triethyl ammonium, monochrome, or TORIMETA Norian ammonium is mentioned as organic ammonium, for example. As an approach of obtaining the carbon black charged in anionic, there is the approach of introducing -COONa into a carbon black front face. Specifically, the method of oxidizing carbon black by sodium hypochlorite is mentioned. Of course, this invention is not necessarily limited to these.

[0014] As a hydrophilic radical combined with the carbon black front face charged in cationicity, the 4th class ammonium is desirable, the next 4th class ammonium is mentioned and that by which these either was combined with the carbon black front face is preferably used as a color material in this invention, for example.

[0015]

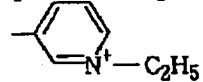
[Formula 4]

 $-\text{NH}_3^+$ 、 $-\text{NR}_3^+$ 、

There is a method of combining N-ethyl ethyl pyridyl radical of the structure shown below, for example as an approach of manufacturing the cationic self-distributed carbon black with which a hydrophilic group which was described above was combined. The method of specifically processing carbon black with a 3-amino-N-ethyl pyridium star's picture is mentioned. Of course, this invention is not limited to this.

[0016]

[Formula 5]



Moreover, in this invention, a hydrophilic radical which was mentioned above may be combined on the surface of carbon black through other groups. The naphthyl group which may have the phenyl group which may have the alkyl group of comparing space and the carbon atomic numbers 1-12 and a substituent, or a substituent as other atomic groups is mentioned. As an example in case the above-mentioned hydrophilic radical joins together on the surface of carbon black through other groups For example, although -CH2 COOM, -C2 H4 COOM, -C3 H6 COOM, -CH2 SO3 M, -C2 H4 SO4 M, -C3 H6 SO4 M, -PhSO3 M, -C5 H10NH3+, etc. are mentioned Of course, this invention is not necessarily limited to these.

[0017] The self-distributed carbon black used for the aquosity black pigment ink of this invention is charged in cationicity or anionic by the hydrophilic radical on the front face of carbon black, and has water-dispersion by repulsion of the ion, and its hydrophilic property is also improving by the hydrophilic radical. Therefore, even if left for a long period of time, the watercolor pigment ink distributed by being stabilized in the aquosity medium is obtained, without the particle size and viscosity of a pigment increasing.

[0018] Moreover, in this invention, the above-mentioned self-distributed carbon black is not limited to one kind, and two or more sorts may be mixed and used for it, and it may adjust a color tone. Moreover,

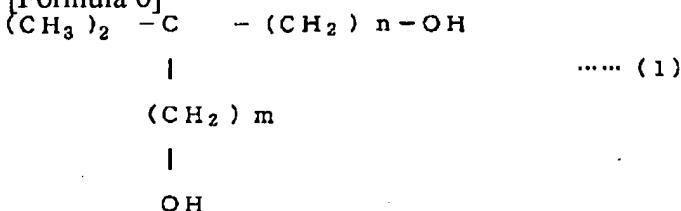
as an addition of the self-distributed carbon black in the pigment ink of this invention, it is 1 - 10% of the weight of the range more preferably 0.1 to 15% of the weight to all ink weight. Furthermore, in addition to self-distributed carbon black, the color tone of ink may be adjusted using a color.

[0019] The black pigment ink of this invention is characterized by the compound of a general formula (1) containing with the self-distributed carbon black used as the above-mentioned color material. as a result of inquiring wholeheartedly so that this invention person may raise the intermittency regurgitation stability of the aquosity black pigment ink containing self-distributed carbon black, when using the compound expressed with a general formula (1), it turned out that intermittent regurgitation stability can be markedly alike and can be raised.

[0020] The compound of a general formula (1) used for the aquosity black pigment ink of this invention is as follows.

[0021]

[Formula 6]



(However, the inside n of a formula expresses the integer of 1-2, and m expresses the integer of 0-2.) Specifically as a compound of the general formula (1) which the aquosity black pigment ink of this invention is made to contain, 3-methyl-1,3-butanediol (a trivial name isoprene glycol or MBD) and 2 and 2-dimethyl 1,3-propanediol (trivial name neopentyl glycol) are used. Moreover, as a content of the compound of a general formula (1), it considers as 1 - 15% of the weight of the range more preferably one to 30% of the weight to the aquosity black pigment ink whole quantity.

[0022] It is desirable to use not the common water containing various ion but deionized water as water which the aquosity black pigment ink of this invention is made to contain. Moreover, as a content of water, it is 50 - 95% of the weight of the range preferably to the aquosity black pigment ink whole quantity.

[0023] Although the aquosity black pigment ink of this invention should just contain the compound and water which are expressed with self-distributed carbon black which was described above, and a general formula (1), a water-soluble organic solvent may be contained.

[0024] As a water-soluble organic solvent, for example Methyl alcohol, ethyl alcohol, N-propyl alcohol, isopropyl alcohol, n-butyl alcohol, sec-butyl alcohol, tert-butyl alcohol, isobutyl alcohol, Alkyl alcohols of the carbon numbers 1-5, such as n-pentanol; Dimethylformamide, Amides, such as dimethylacetamide; Ketones, such as an acetone and diacetone alcohol, or a keto alcohol; tetrahydrofuran, Ether, such as dioxane; A diethylene glycol, triethylene glycol, Tetraethylene glycol, dipropylene glycol, tripropylene glycol, Oxyethylene or oxypropylene copolymers, such as a polyethylene glycol and a polypropylene glycol; Ethylene glycol, Propylene glycol, a trimethylene glycol, triethylene glycol, The alkylene glycol; glycerol with which alkylene groups, such as 1, 2, and 6-hexane triol, contain 2-6 carbon atoms; Trimethylolethane, Trimethylol propane; The ethylene glycol monomethyl (or ethyl) ether, Low-grade alkyl ether, such as the diethylene-glycol monomethyl (or ethyl) ether and the triethylene glycol monomethyl (or ethyl) ether; The triethylene glycol dimethyl (or ethyl) ether, Low-grade dialkyl ether of polyhydric alcohol, such as the tetraethylene glycol dimethyl (or ethyl) ether; Monoethanolamine, Alkanolamines, such as diethanolamine and triethanolamine; a sulfolane, a N-methyl-2-pyrrolidone, 2-pyrrolidone, 1,3-dimethyl-2-imidazolidinone, etc. are mentioned.

[0025] the content of a water-soluble organic solvent which is contained in the ink used by this invention and which was described above -- desirable -- total weight -- it takes more preferably for 2 - 30% of the weight of the range one to 49% of the weight. Moreover, the water-soluble organic solvent

like the above can be used also as mixture, even if independent. In addition, as an amount of the water in the ink used by this invention, it may be about 60 - 90 % of the weight preferably 50 to 98% of the weight.

[0026] Moreover, the ink used by this invention may blend suitably additives other than the above-mentioned component, such as nonionic surfactants, such as a viscosity controlling agent, antiseptics, an anti-oxidant, pH regulator, a defoaming agent, and an ethyleneoxide addition product of an acetone allene glycol, if needed.

[0027] moreover, the range suitable as physical properties of the ink used in this invention -- near 25 degree C -- it is -- pH -- desirable -- 3-12 -- ten to 60 dyn/cm, it is more desirable, 20 - 50 dyn/cm and viscosity are desirable, and the range of 4-10, and 1-30cps of surface tension is 1-10cps more preferably.

[0028] In case the aquosity black pigment ink of this invention is used by ink jet record, it is especially effective. There are a record approach which mechanical energy is made to act on ink and carries out the regurgitation of the drop as the ink jet record approach, and the ink jet record approach which adds heat energy to ink and carries out the regurgitation of the drop by foaming of ink, and especially the aquosity black pigment ink of this invention is suitable for those ink jet record approaches.

[0029] Next, an example of the suitable ink jet recording device of this invention to perform **** record for aquosity black pigment ink of above-mentioned this invention is explained below.

[0030] First, an example of the head configuration which is the principal part using heat energy of an ink jet recording device is shown in drawing 1 and drawing 2.

[0031] Drawing 1 is the sectional view of a head 13 along ink passage, and drawing 2 is a cutting plane Fig. in the A-B line of drawing 1. A head 13 pastes up the glass and the ceramic which have the passage (nozzle) 14 which lets ink pass, silicon or a plastic sheet, and the heater element substrate 15, and is obtained. The heater element substrate 15 consists of a substrate 20 formed with the good ingredient of heat dissipation nature, such as the accumulation layer 19 formed with the exoergic resistor layer 18 formed from refractory materials, such as the electrode 17-1 formed with the protective layer 16 formed with silicon oxide, silicon nitride, carbonization silicon, etc., aluminum, gold, an aluminum-copper alloy, etc. and 17-2, HfB₂, TaN, and TaAl, thermal oxidation silicon, an aluminum oxide, etc., silicon, aluminum and aluminum nitride

[0032] If a pulse-like electrical signal is impressed to the electrode 17-1 of the above-mentioned head 13, and 17-2, the field shown by n of the heater element substrate 15 generates heat quickly, and air bubbles are generated in the ink 21 which is in contact with this front face, and by that pressure, a meniscus 23 will let a projection pass, ink 21 will let the nozzle 14 of a head pass, and it will become the ink globule 24 from discharge and the regurgitation orifice 22, and will fly toward a recorded material 25.

[0033] The external view of an example of a multi-head which put in order many heads shown in drawing 1 is shown in drawing 3. This multi-head pastes up the same exoergic head 28 as the glass plate 27 which has the multi-nozzle 26, and the thing explained to drawing 1, and is made.

[0034] An example of the ink jet recording device which included this head in drawing 4 is shown.

[0035] In drawing 4, 61 is a blade as a wiping member, maintenance immobilization is carried out by the blade attachment component, and the end makes the gestalt of a cantilever. A blade 61 is held with the gestalt which it has been arranged in the location contiguous to the record section by the recording head 65, and was projected in the moving trucking of a recording head 65 in this example.

[0036] 62 is the cap of protrusion **** of a recording head 65, it is arranged at the home position which adjoins a blade 61, moves in the direction perpendicular to the migration direction of a recording head 65, contacts an ink delivery side, and is equipped with the configuration which performs capping. Furthermore, 63 is an ink absorber which adjoins a blade 61 and is formed, and is held like a blade 61 with the gestalt projected in the moving trucking of a recording head 65. The regurgitation recovery section 64 is constituted by the above-mentioned blade 61, cap 62, and the ink absorber 63, and removal of moisture, dust, etc. is performed to a delivery side by a blade 61 and the ink absorber 63.

[0037] The recording head which records on the recorded material which counters the delivery side

which 65 has a regurgitation energy generation means and allotted the delivery by breathing out ink, and 66 are the carriage for carrying a recording head 65 and moving a recording head 65. Carriage 66 engaged with the guide shaft 67 possible [sliding], and has connected a part of carriage 66 with the belt 69 driven by the motor 68 (un-illustrating). Thereby, carriage 66 becomes movable [in alignment with the guide shaft 67], and becomes movable [the record section by the recording head 65, and its adjoining field].

[0038] The feed section for 51 to insert a recorded material and 52 are paper feed rollers driven by the non-illustrated motor. A recorded material is fed to the location which counters with 65 delivery side of a recording head by these configurations, and record is delivered to the delivery unit which arranged the delivery roller 53 along with advance. In case a recording head 65 carries out record termination and returns to a home position in the above configuration, although the cap 62 of the regurgitation recovery section 64 is evacuated from the moving trucking of a recording head 65, the blade 61 is projected in moving trucking. Consequently, wiping of the delivery of a recording head 65 is carried out.

[0039] In addition, when cap 62 performs capping in contact with the regurgitation side of a recording head 65, cap 62 moves so that it may project in the moving trucking of a recording head. When a recording head 65 moves to a recording start location from home PIJISHON, cap 62 and a blade 61 are in the same location as the location at the time of the above-mentioned wiping. Consequently, also in this migration, wiping of the delivery side of a recording head 65 is carried out.

[0040] Migration at the home position of an above-mentioned recording head moves to the home position which adjoined the record section at the predetermined spacing, not only the time of record termination and regurgitation recovery but while moving in a record section for record of a recording head, and the above-mentioned wiping is performed with this migration.

[0041] Drawing 5 is drawing showing an example of the ink cartridge which held the ink supplied to a recording head through ink feed zone material, for example, a tube. 40 is the ink hold section which contained the ink for supply, for example, an ink bag, and the plug 42 made of rubber is formed at the tip here. By inserting a needle (un-illustrating) in this plug 42, supply of the ink in the ink bag 40 on a head is enabled. 44 is an ink absorber which receives waste ink.

[0042] That in which the liquid-facing surface with ink is formed with polyolefine, especially polyethylene as the ink hold section is desirable.

[0043] It is used suitable not only for that from which the head and the ink cartridge became another object as mentioned above but the thing with which they as show drawing 6 were united as an ink jet recording apparatus used by this invention. In drawing 6 , 70 is a record unit, the ink hold section which held ink into this, for example, an ink absorber, is contained, and the ink in this ink absorber has composition breathed out as an ink droplet from the head section 71 which has two or more orifices. It is desirable for this invention to use polyurethane as an ingredient of an ink absorber.

[0044] Moreover, structure which is the ink bag with which the ink hold section taught the spring etc. to the interior not using the ink absorber is sufficient.

[0045] 72 is atmospheric-air free passage opening for making atmospheric air open the interior of a cartridge for free passage. This record unit 70 is changed and used for the recording head 65 shown in drawing 4 , and attachment and detachment of it are attained to carriage 66.

[0046] Next, it can have ink which fills the perimeter of the nozzle formation substrate which has two or more nozzles, the pressure generating component which consists of piezoelectric material which counters a nozzle and is arranged, and an electrical conducting material, and this pressure generating component as a desirable example using mechanical energy of an ink jet recording device, the variation rate of the pressure generating component can be carried out with applied voltage, and the on-demand ink jet recording head which makes the small drop of ink breathe out from a nozzle can be mentioned. An example of the configuration of the recording head which is the principal part of the recording device is shown in drawing 7 .

[0047] It is joined to the ink passage 80 which was open for free passage in the ink room (un-illustrating), the orifice plate 81 for carrying out the regurgitation of the ink droplet of the desired volume, the diaphragm 82 which makes a direct pressure act on ink, and this diaphragm 82, and the head

consists of a piezoelectric device 83 displaced with an electrical signal, and a substrate 84 for carrying out directions immobilization of an orifice plate 81 and the diaphragm 82 grade.

[0048] In drawing 7, the ink passage 80 is formed with a photopolymer etc., a delivery 85 is formed by punching according [an orifice plate 81] metals, such as stainless steel and nickel, to electrocasting or press working of sheet metal etc., a diaphragm 82 is formed with a metal film, high elasticity resin films, etc., such as stainless steel, nickel, and titanium, and a piezoelectric device 83 is formed with dielectric materials, such as barium titanate and PZT.

[0049] The recording head of the above configurations gives a pulse-like electrical potential difference to a piezoelectric device 83, generates distortion stress, and is made to transform the diaphragm with which the energy was joined to the piezoelectric device 83, and it operates so that it may record by pressurizing the ink in the ink passage 80 perpendicularly, and breathing out an ink droplet (un-illustrating) from the delivery 85 of an orifice plate 81.

[0050] Such a recording head is used including in the same ink jet recording device as what was shown in drawing 4. Actuation of the details of an ink jet recording device is not performed like point **, and does not interfere.

[0051]

[Example] Next, this invention is not limited by the following example, although an example and the example of a comparison are given and this invention is explained more concretely. In addition, as long as there is no notice especially, there are weight criteria among a sentence with the "section" and "%."

[0052] Acid carbon black "MA-77" (pH3, Mitsubishi Chemical make) which is example 1 (production of pigment dispersion liquid 1) marketing After often mixing 300g in 1,000ml of water, 450g (12% of available chlorine concentration) of sodium hypochlorite was dropped at this, and it stirred at 100-105 degrees C for 10 hours. The obtained slurry was filtered by Toyo Roshi No.2 (product made from ADOBANTISU), and the pigment particle was fully rinsed. This pigment wet cake was re-distributed in 3,000ml of water, and it desalted by the reverse osmotic membrane to 0.2 microseconds of electric conductivity. Furthermore, these pigment dispersion liquid (pH=8-10) were condensed to 10 % of the weight of pigment concentration. By the above approach, the pigment dispersion liquid 1 with which -COONa radical was combined on the surface of carbon black and by which anionic self-distributed carbon black was distributed were obtained.

[0053] The component below ink creation of an example 1 was mixed, after stirring enough and making it dissolve, pressure filtration was carried out in the pore size 3.0micrometer microfilter (Fuji Photo Film make), and the ink of an example 1 was adjusted.

[0054]

- Pigment dispersion liquid 1 The 30 sections -3-methyl 1,3-butanediol The ten sections - water Each component shown in 60 section examples 2 - 6 following was mixed, after stirring enough and making it dissolve, pressure filtration was carried out in the pore size 3.0micrometer microfilter (Fuji Photo Film make), and the ink of examples 2-5 was adjusted, respectively.

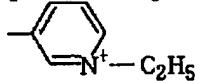
[0055]

Ink presentation of an example 2 - pigment dispersion liquid 1 The 30 sections -2 and 2-dimethyl-1,3-propanediol The ten sections - water The 60 sections The ink presentation of an example 3 - Pigment dispersion liquid 1 The 30 sections -3-MECHIRU 1,3-butanediol The ten sections - thiodiglycol The five sections - isopropyl alcohol The three sections - water The 52 sections Ink presentation of an example 4 - pigment dispersion liquid 1 The 30 sections -3-methyl-1,3-butanediol The seven sections - glycerol The seven sections - acetylene glycol ethyleneoxide addition product (trade name: ASECHIRE Norian EH, Kawaken Fine Chemicals make) The 0.15 sections - water The 55.85 sections Ink presentation of an example 5 - pigment dispersion liquid 1 The 30 sections -2 and 2-dimethyl-1,3-propanediol The five sections - diethylene glycol The five sections - glycerol The five sections - acetylene glycol ethyleneoxide addition product (trade name: ASECHIRE Norian EH, Kawaken Fine Chemicals make) The 0.15 sections - water DBP oil absorption by 230m2 / g Carbon black 10g of 70m2 / g, [54.85 section examples 6 (production of pigment dispersion liquid 2) surface area] After often mixing 3-amino-N-ethyl pyridinium star's picture 3.06g in 72g of water, 1.62g of nitric acids was

dropped at this, and it stirred at 70 degrees C. The solution which melted the 1.07g sodium nitrite was added to 5g water after several minutes, and it stirred for further 1 hour. The obtained slurry was filtered by Toyo Roshi No.2 (product made from ADOBANTISU), and the pigment particle was fully rinsed. It re-distributed in 3,000ml of water, and this pigment wet cake was desalinated by the reverse osmotic membrane to 0.2 microseconds of electric conductivity. Furthermore, these pigment dispersion liquid were condensed to 10 % of the weight of pigment concentration. The pigment dispersion liquid 2 by which the cationic self-distributed carbon black by which the following hydrophilic radical was combined with the front face of carbon black was distributed by the above approach were obtained.

[0056]

[Formula 7]



The component below ink creation of an example 6 was mixed, after stirring enough and making it dissolve, pressure filtration was carried out in the pore size 3.0micrometer microfilter (Fuji Photo Film make), and ink 6 was adjusted.

[0057]

- Pigment dispersion liquid 2 The 30 sections -3-methyl 1,3-butanediol The ten sections - water Each component shown in 60 section examples 7 - 10 following was mixed, after stirring enough and making it dissolve, pressure filtration was carried out in the pore size 3.0micrometer microfilter (Fuji Photo Film make), and the ink of examples 7-10 was adjusted, respectively.

[0058]

Ink presentation of an example 7 - pigment dispersion liquid 2 The 30 sections -2 and 2-dimethyl-1,3-propanediol The ten sections - water The 60 sections Ink presentation of an example 8 - pigment dispersion liquid 2 The 30 sections -3-MECHIRU 1,3-butanediol The ten sections - diethylene glycol The five sections - isopropyl alcohol The three sections - water The 52 sections Ink presentation of an example 9 - pigment dispersion liquid 2 The 30 sections -3-methyl-1,3-butanediol The seven sections - glycerol The seven sections - acetylene glycol ethyleneoxide addition product (trade name: ASECHIRE Norian EH, Kawaken Fine Chemicals make) The 0.15 sections - water The 55.85 sections Ink presentation of an example 10 - pigment dispersion liquid 2 The 30 sections -2 and 2-dimethyl-1,3-propanediol The five sections - diethylene glycol The five sections - glycerol The five sections - acetylene glycol ethyleneoxide addition product (trade name: ASECHIRE Norian EH, Kawaken Fine Chemicals make) The 0.15 sections - water Each component shown in the example 1 of a 54.85 section comparison - 8 following was mixed, after stirring enough and making it dissolve, pressure filtration was carried out in the pore size 3.0micrometer microfilter (Fuji Photo Film make), and the ink of the examples 1-10 of a comparison was adjusted, respectively.

[0059]

Ink presentation of the example 1 of a comparison - pigment dispersion liquid 1 The 30 sections - water The 70 sections Ink presentation of the example 2 of a comparison - pigment dispersion liquid 1 The 30 sections - thiodiglycol The five sections - isopropyl alcohol The three sections - water The 62 sections Ink presentation of the example 3 of a comparison - pigment dispersion liquid 1 The 30 sections - glycerol The seven sections - acetylene glycol ethyleneoxide addition product (trade name: ASECHIRE Norian EH, Kawaken Fine Chemicals make) The 0.15 sections - water The 62.85 sections An ink presentation and pigment dispersion liquid 1 of the example 4 of a comparison The 30 sections - diethylene glycol The five sections - glycerol The five sections - acetylene glycol ethyleneoxide addition product (trade name: ASECHIRE Norian EH, Kawaken Fine Chemicals make) The 0.15 sections - water The 59.85 sections Ink presentation of the example 5 of a comparison The - pigment dispersion-liquid 2 30 section - water The 70 sections Ink presentation of the example 6 of a comparison - pigment dispersion liquid 2 The 30 sections - diethylene glycol The five sections - isopropyl alcohol The three sections - water The 62 sections Ink presentation of the example 7 of a comparison - pigment dispersion

liquid 2 The 30 sections - glycerol The seven sections - acetylene glycol ethyleneoxide addition product (trade name: ASECHIRE Norian EH, Kawaken Fine Chemicals make) The 0.15 sections - water The 62.85 sections Ink presentation of the example 8 of a comparison - pigment dispersion liquid 2 The 30 sections - diethylene glycol The five sections - glycerol The five sections - acetylene glycol ethyleneoxide addition product (trade name: ASECHIRE Norian EH, Kawaken Fine Chemicals make) The 0.15 sections - water The 59.85 sections [evaluation] The ink of the above-mentioned examples 1-10 and the examples 1-8 of a comparison is used. The following evaluation was performed using ink jet recording device BJC-4200 (Canon, Inc. make) which has the on-demand mold multi-recording head which makes ink breathe out by giving the heat energy according to a record signal to ink. The result is shown in Table 1.

- Stability **-10-second continuation regurgitation of the intermittent regurgitation -> since it was decided by the quiescent time of ** whether turbulence of a discharge direction will occur in the regurgitation of the beginning of ** when the intermittent control action of the pause ->** continuation regurgitation above was performed during ** 1 scheduled time, by changing this time amount gradually, the stability of the intermittent regurgitation was measured and the following criteria estimated. In addition, evaluation was performed at the environmental temperature of 5 degrees C, and 10% of humidity.

[0060] O : even if it stopped 31 seconds or more, it breathed out to stability.

[0061] O : even if it stopped for 21 to 30 seconds, it breathed out to stability.

[0062] x: The regurgitation was carried out to stability only by the quiescent time not more than 20 second.

[0063]

[Table 1]

表1:評価結果

	間欠吐出の安定性
実施例1	○
実施例2	○
実施例3	○
実施例4	◎
実施例5	◎
実施例6	○
実施例7	○
実施例8	○
実施例9	◎
実施例10	◎
比較例1	×
比較例2	×
比較例3	×
比較例4	×
比較例5	×
比較例6	×
比較例7	×
比較例8	×

[0064]

[Effect of the Invention] As explained above, according to this invention, the ink jet record approach and ink jet recording device using aquosity black pigment ink excellent in the regurgitation stability after a printing pause and the aquosity black pigment ink to apply are offered.

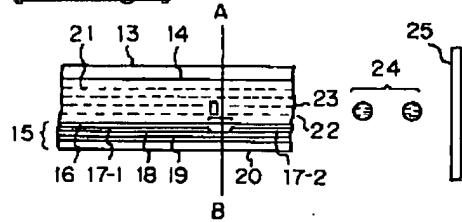
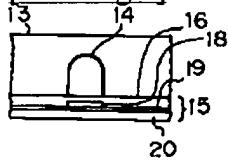
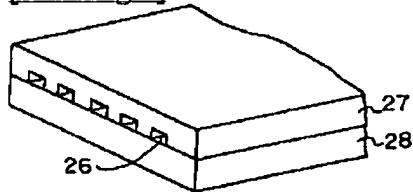
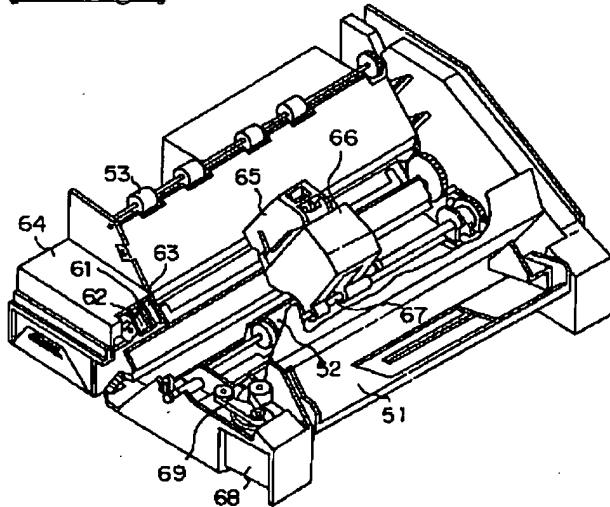
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*** NOTICES ***

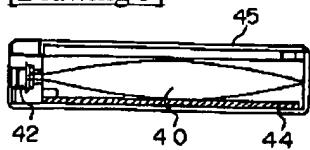
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3. In the drawings, any words are not translated.

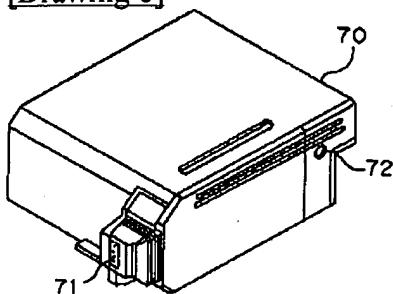
DRAWINGS

[Drawing 1]**[Drawing 2]****[Drawing 3]****[Drawing 4]**

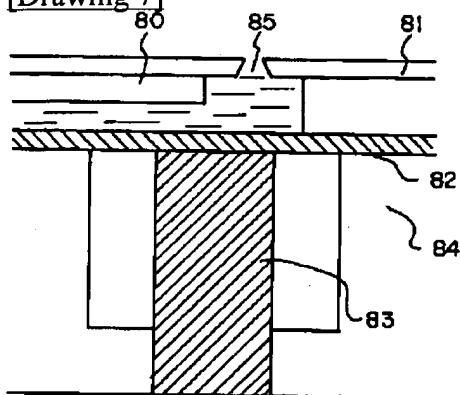
[Drawing 5]



[Drawing 6]



[Drawing 7]



[Translation done.]